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(54) **Analyser detector window**

(57) The detector window for an analyser, particularly an X-ray analyser, is a thin film, capable of withstanding a required pressure difference, with a thickness of 0.5 µm. Preferably the thin film is made of polyimide, with or without glass fibres, is manufactured by means of photolithography, and may have a diameter of 0 - 150mm. The film may have a 30Å thick aluminium coating and may also have a 0.1-0.2 µm thick layer of diamond powder, boron nitride or boron carbide to render the film gas-proof. The window is permeable to soft X-rays.

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ANALYSER DETECTOR WINDOW AND A METHOD FOR MANUFACTURING THE SAME

The present invention relates to the detector window of an X-ray analyser, through which window the intensity formed by soft X-rays is measured. The invention also relates to a method for manufacturing the detector window.

Traditionally the window of an X-ray analyser has been made of beryllium. This kind of window is necessary when the detector is not placed in a vacuum, as is the case with a scanning electron microscope, although the inner components of the apparatus are located in a vacuum. Owing to the low molar mass of beryllium, the detector window must, however, be at least 7 μm thick in order to create a sufficient twisting and mechanical strength.

In order to make the detector window of an X-ray analyser thinner and thus better in operation, plastic materials have also been used in the production of detector windows. US patent 4,119,234 describes a vacuum-tight window made of plastic, such as polyimide. In the article X-γ-β ray detector windows of composite material replacing beryllium in the 4.2 - 420 K temperature range by Rimbert J.N. and Testard O.A., Nuclear instruments and Methods in Physics Research A 251 (1986), p. 95-100, the beryllium windows are replaced by windows formed of aluminium layers fitted in a laminated fashion between aligned polyimide

membranes. Furthermore, from US patent 4,061,944 it is known to use membranes of polymers known by the trademarks Kapton or Mylar in the making of windows for electron beam generators.

US patent 3,262,002 introduces an X-ray detector where the windows are manufactured of various different materials such as nitrocellulose. Nitrocellulose has also been used in the electron microscope of the US patent 2,241,432, comprising a window with a small area, which window can, however, be used in connection to a pressure difference of one atmosphere. This window is formed as a colloid containing nitrocellulose, while the window thickness is within the range of 0.1 - 1.0 μm.

US patent 3,319,064 relates to a slidable window system for an X-ray analyser, wherein three windows are grouped together to be operated so that only two of the windows are operated simultaneously, and so that they are interchangeable with two beryllium windows which prevent any pressure difference between the internal and external parts of the apparatus. Moreover, the window system includes one beryllium and one colloid window, which are insulated, due to the pressure difference, by means of the two preceding windows.

The purpose of the present invention is to realize an improved detector window for an analyser for analysing Z-rays, particularly soft X-rays, which window is made of a

thin polymer film and which endures the pressure difference between the internal external parts of the analyser without a specific protective structure. The detector window of the invention is a thin film with the thickness of 0.5 μm.

The X-ray analyser detector window of the present invention is made, by making use of photolithography, of polymer products sold under the trademarks PYRALIN or KAPTON. The PYRALIN product is composed, according to The Encyclopaedia of Chemical Trademarks and Synonyms Vol. III, of polyimide and glass fiber, whereas the KAPTON product, according to the Thesaurus of Chemical Products Vol. II, is a polyimide membrane. Particularly the polymer products PYRALIN PI 2555 and PYRALIN PI 2556 are well suited to the method of the present invention.

In order to manufacture the detector window of the X-ray analyser of the present invention by means of photolithography, the required 25 μm thick metal plate is advantageously made of, for example, copper or copper alloy, such as brass, of wolfram, nickel or gold. In the beginning of the production process, the metal plate is subjected to supersonic cleaning by means of Freon, whereafter the plate is washed by distilled water. The cleaned plate is then dried by blowing with an inert gas such as nitrogen, by heating the plate momentarily up to the temperature of 90°C. Onto the dried plate there is then applied, in order to improve the sticking of the polymer product proper, a layer

of for instance silane, whereafter the polymer product forming the X-ray analyser detector window of the invention can be spread onto the plate. Prior to the spreading of the polymer product, it is possible, if desired, to apply a thin layer with the thickness of 0.1-0.2 μm, made of diamond powder, boron nitride or boron carbide, in which case the final film is made gas-proof, for instance helium-proof.

The film material applied on the metal plate is further dried at the temperature of 350-370^{o}C in a nitrogen atmosphere. Thereafter the plate, serving as the mask, is imaged, and the obtained image is etched off, for instance by means of ferric chloride. The remaining product is a metal-framed polymer film with the thickness of 0.5 μm, suited to be used as a window. Because this detector window made by means of photolithography is permeable to visible light, the window is treated to make it impermeable to visible light. The treatment is carried out by applying onto at least one window surface a thin aluminium layer with the thickness of roughly 30 x 10^{-10}m (=30 Angströms).

The X-ray analyser detector window manufactured according to the method of the present invention is advantageously suited to transmit and/or receive soft X-rays, the energy whereof is within the range 100-1000 eV. Moreover, the detector window allows for a pressure difference larger than one atmosphere in between the interior parts of the analyser and the environment. Thus the detector

window can be used for example when the pressure inside the analyser essentially corresponds to that of a vacuum, and the pressure in the exterior is one atmosphere, or even in an opposite case, when a gas pressure is formed inside the analyser, and the analyser itself is located in a vacuum. The detector window can also be used in circumstances where the pressure difference is below one atmosphere, or even when the pressure is equal on both sides of the window.

The material used in the detector used in the detector window of the invention, which material contains polyimide or polyimide and glass fiber, is chemically inert and harmless to X-rays. Moreover, the detector window of the invention can be used in relatively high temperatures, up to the range of 300-350^{o}C. Furthermore, the method of the invention enables the production of a large detector window with a diameter of even 150 mm.

CLAIMS

1. A detector window for an analyser, more particularly an X-ray analyser, for the penetration of soft X-rays, when the detector window is at least on one surface in contact with a pressure equal to that of a vacuum, in the form of a thin film with a thickness of 0.5 μm.

2. The detector window of claim 1, in which the detector window is made of polymer.

3. The detector window of claim 2, in which the detector window is made of polyimide.

4. The detector window of claim 1, in which the detector window is made of polyimide and glass fiber.

5. The detector window of any of the claims 1-4, having a diameter of the detector window between 0-150 mm.

6. A method for manufacturing the detector window of any of claims 1-5, in which the detector window is made by means of photolithography.

7. The method of claim 6, in which on at least on surface of the detector window there is formed an aluminium layer with a thickness about 30 Angströms in order to make it impermeable to visible light.

8. The method of claim 6 or 7, in which on at least one surface of the detector window there is formed a gas-proof film.

9. A detector window for an analyser substantially as herein before described.